THE PREPARATION OF RGU-1

by

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ABSTRACT

The Canadian Certified Reference Materials Project has prepared a uranium reference material on behalf of the International Atomic Energy Agency by dilution of 2.84 kg of BL-5 with 500 kg of floated silica. Homogeneity of this material, encoded RGU-1, was confirmed by an X-ray fluorescence procedure at CANMET and by a neutron activation procedure by Chemex Laboratories Ltd., North Vancouver, British Columbia. The concentration of uranium in RGU-1 was calculated to be 400 µg/g with an uncertainty of 2.1 µg/g. Uranium results from CANMET and Chemex were confirmatory.

INTRODUCTION

The Canadian Certified Reference Materials Project (CCRMP) agreed in 1984 to prepare a uranium reference material, RGU-1, on behalf of the International Atomic Energy Agency, IAEA. The work was accepted by the Canada Centre of Mineral and Energy Technology as Cost Recovery Job No. 025202.

RGU-1 is intended for use in calibrating laboratory gamma-ray spectrometers to determine their sensitivities and stripping ratios for determination of U, Th and K ratios. This reference material was to be prepared by dilution of uranium reference ore BL-5 (1) of CCRMP (7.09% U) with a silica matrix of negligible U and Th content to give a final U concentration of 400 ± 8 µg/g. Since the advent of the preparation of RGU-1, BL-5 has been certified for radium-226 (2) and lead-210 (3), thereby confirming BL-5 to be in secular equilibrium. This report describes the physical and chemical procedures by which RGU-1 was prepared and characterized.

PREPARATION AND NATURE

RAW MATERIALS

The characterization and certification of BL-5 are described elsewhere (1-3) - Certificate of Analysis attached. The most important constituent parameters are reviewed in Table 1.

Table 1 - Values of constituents of BL-5

Constituent	Concentration
U	7.09 ± 0.03%*
Ra-226	857 ± 38 Bq/g*
Pb-210	866 ± 21 Bq/g*
Th	40 µg/g
K	0.4%

*Certified value

The silica diluent (500 kg) was purchased from Fisher Scientific, catalogue #S-152. It is a floated powder of approximately 240 mesh size. A particle size analysis is reported in Table 2.

Table 2 - Particle size analysis (wet screen)

Size of fraction (μm)		Silica	RGU-1*
		mass %	
	+104	0.0	0.0
-104	+ 74	8.3	8.3
- 74	+ 46	19.9	19.8
- 46	+ 37	14.0	13.9
- 37		57.8	58.0

*Calculated from corresponding values of silica and BL-5 (1).

The silica was investigated at CANMET by gamma-ray spectrometry and the equivalent uranium and equivalent thorium values were estimated to be 0.2 ± 0.1 and 0.4 ± 0.3 μg/g, respectively. The silica contribution to the overall uranium content of RGU-1 (~400 μg/g) is therefore negligible. On the other hand, the equivalent thorium value of the silica is approximately the same as the 0.2 μg/g Th value expected for RGU-1 from BL-5 (0.004% Th) but does not affect significantly the favorable U:Th ratio required in RGU-1.

The silica was also analyzed by a semi-quantitative optical emission technique.

PHYSICAL PREPARATION

RGU-1 was prepared in two batches because of equipment limitations. For one batch, 245.8 kg of silica were blended with 1.395 kg of BL-5 for 8 h in a 570-L blender. The other batch consisted of 253.8 kg of silica and 1.440 kg BL-5 and was treated similarly. These batches were divided approximately in-half and each portion of one batch was blended with a portion of the other batch for 3 h, thereby giving two new batches. This procedure was repeated twice more to ensure that the two batches obtained at the end were homogeneous. RGU-1 was bottled in 500-g lots in plastic bottles supplied by IAEA. These bottles had narrow necks intended for liquid storage and as a

consequence gave considerable difficulty in the filling process. Material losses were much greater than expected and only 972 instead of 1000 bottles could be provided.

HOMOGENEITY CONFIRMATION

The homogeneity of RGU-1 was assessed by CANMET by an X-ray fluorescence technique and by Chemex Laboratories Ltd., North Vancouver, British Columbia (P.O. No. 4.5277) by a neutron activation technique by analyzing in triplicate for uranium 16 bottles selected from the stock of 972. The stock was divided into 15 lots of 62 bottles and a 16th lot of 42 bottles. The code number of the bottle from the first lot was selected at random out of 1 to 62. The code number of the remaining 15 bottles was given by the code number of the preceding bottle plus 62. Chemex Laboratories Ltd., were sent three 2-g samples from each bottle; the samples were coded accordingly to a random selection scheme.

A one-way analysis of variance technique was used to assess the homogeneity (4). Herein the ratio of the between-bottles to within-bottle mean square is compared to the F statistic at the 95% level of probability. The results for the evaluation by CANMET and by Chemex Laboratories are shown in Tables 3 and 4. No evidence for between-bottles inhomogeneity was detected by either CANMET or Chemex Laboratories Ltd. Another manner of evaluating the homogeneity results is reported in Table 5. Herein the mean of bottles 19 to and including 453 from one batch is compared with that for bottles 515 to and including 949 of the other batch of fully blended material. The observed difference between the mean values is not statistically significant in view of the values of σ, the standard deviation of the means.

NATURE OF RGU-1

The approximate chemical composition of RGU-1 shown in Table 6 is based on the composition of BL-5 and the semi-quantitative optical emission analyses of the silica diluent and RGU-1. The calculated particle size distribution is reported in Table 2.

The uranium concentration of RGU-1 calculated solely on the ratio of silica to BL-5 is 400 µg/g with 95% confidence intervals of ± 1.7 µg/g. The latter reflects only the estimated uncertainty in the measurement of uranium in BL-5. The potential error in the weighing of the silica was estimated to

Table 3 - Confirmation of homogeneity of RGU-1 by CANMET

Bottle No.	Counts Individual			Mean
19	1097	1078	1064	1079.7
81	1084	1087	1093	1088.0
143	1065	1086	1098	1083.0
203	1052	1078	1086	1072.0
268	1099	1097	1082	1092.7
329	1090	1071	1074	1078.3
391	1075	1078	1070	1074.3
453	1081	1076	1058	1071.7
515	1081	1075	1071	1075.7
577	1079	1082	1069	1076.7
639	1072	1062	1088	1074.0
701	1090	1076	1077	1081.0
763	1054	1066	1053	1057.7
825	1065	1070	1089	1074.7
887	1095	1077	1085	1085.7
949	1067	1064	1074	1068.3
		Overall mean is		1077.1

Analysis of Variance Table

Source of Variation	D.F.	Sum of squares	Mean squares
Between-sets	15	3.084×10^3	2.056×10^2
Within-sets	32	3.737×10^3	1.168×10^2
Total	47	6.822×10^3	

Calculated F statistic = 1.761

F.95(15,32) = 1.992

Null hypothesis of no difference between bottles is accepted for uranium

Table 4 - Confirmation of homogeneity of RGU-1 by Chemex

Bottle No.	Mass % U			
	Individual			Mean
19	.0390	.0390	.0382	.0387
81	.0390	.0390	.0390	.0390
143	.0390	.0382	.0382	.0384
205	.0390	.0382	.0390	.0387
267	.0407	.0382	.0399	.0396
329	.0407	.0382	.0382	.0390
391	.0390	.0399	.0399	.0396
453	.0382	.0399	.0373	.0384
515	.0390	.0390	.0399	.0393
577	.0390	.0390	.0399	.0393
639	.0399	.0390	.0390	.0393
701	.0399	.0390	.0390	.0393
763	.0390	.0390	.0416	.0399
825	.0382	.0382	.0390	.0384
887	.0399	.0390	.0382	.0390
949	.0390	.0399	.0390	.0393
		Overall mean is		.0391

Analysis of Variance Table

Source of Variation	D.F.	Sum of squares	Mean squares
Between-sets	15	8.390×10^{-6}	5.593×10^{-1}
Within-sets	32	2.157×10^{-5}	6.742×10^{-1}
Total	47	2.996×10^{-5}	

Calculated F statistic = 0.830

F.95(15,32) = 1.992

Null hypothesis of no difference between bottles is accepted for uranium

Table 5 - Comparison of means of the two batches of RGU-1

Laboratory	Batch	Bottles	Mean ± σ
CANMET	1	19 - 453	1080.0 ± 7.6 counts
	2	515 - 989	1074.2 ± 8.4 counts
Chemex	1	19 - 453	389 ± 4.7 µg/g U
	2	515 - 989	392 ± 4.2 µg/g U

Table 6 - Approximate chemical composition of RGU-1

Element	Mass %
Si	46.0
U	0.08
Al	0.03
Fe	0.03
Ca	0.02
Na	0.02
C,total	0.01
Mg	0.008
Pb	0.008
K	0.002
Ti	0.002
S	0.002
Th	< 1 µg/g

be 1.45 kg for the total mass of 500 kg leading to an uncertainty of ± 1.2 µg/g U. Errors in the weighing of BL-5 were assumed to be negligible. The total uncertainty of the concentration of uranium is composed of the uncertainty of the measurement of uranium in BL-5 and the uncertainty in the dilution by weighing and was estimated to be ± 2.1 µg/g. Therefore:

RGU-1 400 ± 2.1 µg/g U

Analysis of RGU-1 at CANMET by an X-ray fluorescence technique found the concentration of uranium to be 0.041%. The mean from Chemex Laboratories Ltd. was 0.0391% and the overall mean from both laboratories is 0.040%.

REFERENCES

1. Faye, G.H., Bowman, W.S. and Sutarno, R. "Uranium ore BL-5 - A Certified Reference Material"; CANMET Report 79-4; CANMET, Energy, Mines and Resources Canada; 1979.

2. Smith, C.W. and Steger, H.F. "Radium-226 in certified uranium reference ores DL-1a, BL-4a, DH-1a and BL-5"; CANMET Report 83-9E; CANMET, Energy, Mines and Resources Canada; 1983.

3. Smith, C.W. and Steger, H.F. "Lead-210 in certified uranium reference ores DL-1a, BL-4a, DH-1a and BL-5"; CANMET Report 84-10E; CANMET, Energy, Mines and Resources Canada; 1984.

4. Brownlee, K.A. "Statistical theory and methodology in science and engineering"; John Wiley and Sons, Inc.; New York; 1960.

CANMET CONTRIBUTERS

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